occupancy of $\frac{1}{3}$, has better hydrogen bonds than $W(1)$ itself (Table 9, D). No attempt has been made to indicate donors and acceptors in the hydrogen bonding, because the hydrogen atoms were not located and several bonding schemes are feasible. The large temperature motion of the water molecules (Table 1) is probably the reason that only few reflections with $2\theta > 90°$ could be observed. All other intermolecular distances less than 3·50 Å are shown in Table 9, E.

In summary it is interesting to note that the structure is sufficiently large and complicated for it to be possible to recognize so called hydrophilic and hydrophobic regions.

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The Crystal and Molecular Structure of 1,8-Dinitrosonaphthalene

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1,8-Dinitrosonaphthalene crystallizes in space group $P2_1/b$ with $a=13{\cdot}13$, $b=16{\cdot}01$, $c=3{\cdot}89$ Å, $=92{\cdot}7°$ and with $Z=4$. The structure was determined from photographic X-ray data and refined by the least-squares method to $R=0{\cdot}078$ for 1009 reflexions. The molecule is in the form of an internal nitroso dimer with an N–N bond 1·38 Å long. The naphthalene nucleus shows similar distortions to those found in the acenaphthenes.

Introduction

It is possible to devise a number of plausible molecular structures for 1,8-dinitrosonaphthalene. Data from chemical and spectroscopic measurements do not allow an unambiguous distinction between these structures. Therefore the structure of the crystal was determined by X-ray diffraction.

Experimental

Red-brown crystals of the compound were supplied by Professor M. C. Whiting of Bristol University (Whiting, 1969). The crystal data are: $C_{10}H_6N_2O_2$, $M=186{\cdot}2$, monoclinic,

$a=13{\cdot}13\pm0{\cdot}03$, $b=16{\cdot}01\pm0{\cdot}03$, $c=3{\cdot}89\pm0{\cdot}01$ Å,
$\alpha=92{\cdot}7\pm0{\cdot}3°$,
$Z=4$,
$D_m=1{\cdot}51$ g. cm^{-3} by flotation,
$D_c=1{\cdot}513$ g. cm^{-3} Cu $K\alpha$ 1·5418 Å,
$\mu=9{\cdot}21$ cm^{-1}.

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Systematic extinctions $0kl$, $k=2n+1$ and $h00$, $h=2n+1$; space group $P2_1/b$ (C^5_{2h}, No. 14).

From a crystal sealed in a glass capillary equi-inclination Weissenberg photography with Cu $K\alpha$ radiation of the $hk0$ to $hk3$ layers produced 1009 observable reflexions. The visually estimated data were corrected for Lorentz and polarization effects but not for absorption.

Solution and refinement of the structure

The structure was solved by a symbolic addition process. The data were one of several sets of data used in the testing and development of a symbolic addition system now in extensive use in our laboratory (Hodder, Prout & Rollett, 1970). The final version of the program used all triple products of probability over 0·9 given by the 400 reflexions with $E>0{\cdot}95$. Perhaps the most important feature of the system is that the number and assignment of the symbolic phases, and the origin, are determined by the computer and not by the operator. The reflexions $21\bar{1}$, $23\bar{2}$ and 11,2,1 were assigned positive signs to fix the origin of the unit-cell, the sign of 10,0,0 was found to be positive by $\sum_1$ relations and the signs of 991 and 3,13,1 were represented by the symbols A and B. 184 signs were determined with probabilities of more than 0·9975. The probability limit was then reduced to 0·982 and a further 41 signs were determined. The most consistent solution with $A\equiv-$ and $B\equiv-$ was the correct one and gave a set of signed E's from which a Fourier synthesis was computed. The synthesis had 14 large peaks corresponding to the non-hydrogen atoms of the molecule (I). The trial structure was refined by the full-matrix least-squares method first assuming isotropic and then anisotropic thermal motion. The layer scale factors were revised in the isotropic but not in the anisotropic refinement. The hydrogen atoms were located from a difference synthesis after 4 cycles of isotropic refinement but their coordinates were deduced from the molecular geometry. The refinement was then continued anisotropically for a further 5 cycles and converged at an $R\ (=\sum |F_o| - |F_c|/\sum|F_o|)=0{\cdot}078$. The weighting scheme was $w=1/(1+[(F_o-P_1)/P_2]^2)$ where $P_1=P_2=12$ on the scale of Table 2.

The observed structure amplitudes and calculated structure factors calculated from the final atomic parameters in Table 1 are given in Table 2. These results

Table 1. *Fractional atomic coordinates* ($\times 10^4$) *and thermal parameters* ($\times 10^4$) *with standard deviations in parenthesis*

	x/a	y/b	z/c	U_{iso}
C(1)	3342 (3)	4337 (3)	8361 (11)	–
C(2)	3619 (4)	5154 (3)	9230 (12)	–
C(3)	2861 (4)	5651 (3)	10988 (12)	–
C(4)	1897 (4)	5353 (3)	11831 (11)	–
C(5)	673 (3)	4070 (3)	11252 (12)	–
C(6)	560 (4)	3252 (4)	9945 (14)	–
C(7)	1345 (4)	2797 (3)	8137 (13)	–
C(8)	2257 (3)	3212 (3)	7843 (11)	–
C(9)	2376 (3)	4036 (3)	9166 (10)	–
C(10)	1618 (3)	4513 (3)	10843 (11)	–
N(1)	3848 (3)	3668 (2)	6490 (10)	–
N(2)	3203 (3)	2991 (2)	6169 (10)	–
O(1)	4738 (3)	3656 (2)	5181 (10)	–
O(2)	3443 (3)	2323 (2)	4550 (9)	–
H(1)	4370	5397	8591	500
H(2)	3044	6303	11650	500
H(3)	1509	5845	13347	500
H(4)	−41	4248	12528	500
H(5)	−168	2945	10272	500
H(6)	1237	2157	7058	500

	U_{11}	U_{22}	U_{33}	$2U_{23}$	$2U_{31}$	$2U_{12}$
C(1)	421 (23)	453 (26)	429 (27)	−19 (39)	−91 (37)	115 (38)
C(2)	489 (25)	520 (28)	566 (31)	47 (45)	−108 (41)	−103 (42)
C(3)	630 (30)	437 (28)	574 (32)	−47 (45)	−146 (46)	−44 (45)
C(4)	590 (28)	499 (28)	446 (29)	−151 (42)	−50 (42)	222 (45)
C(5)	446 (25)	795 (38)	475 (31)	237 (50)	9 (41)	33 (48)
C(6)	528 (29)	767 (40)	627 (35)	313 (56)	−112 (47)	−208 (52)
C(7)	591 (29)	574 (31)	559 (31)	220 (47)	−255 (47)	−149 (48)
C(8)	478 (24)	440 (26)	382 (26)	68 (39)	−120 (37)	46 (40)
C(9)	419 (22)	453 (26)	347 (25)	14 (38)	−134 (34)	31 (37)
C(10)	473 (25)	587 (30)	359 (26)	72 (41)	−71 (37)	142 (42)
N(1)	456 (21)	572 (24)	560 (26)	−2 (38)	64 (35)	204 (37)
N(2)	532 (22)	439 (22)	553 (25)	−140 (36)	−88 (36)	129 (36)
O(1)	486 (20)	800 (26)	864 (27)	62 (39)	433 (36)	282 (36)
O(2)	865 (27)	590 (22)	756 (26)	−464 (37)	−90 (39)	386 (38)

The temperature factor has the form:

$$\exp\,[-2\pi^2(U_{11}h^2a^{*2}+U_{22}k^2b^{*2}+U_{33}l^2c^{*2}+2U_{23}klb^*c^*+2U_{31}lhc^*a^*+2U_{12}hka^*b^*)].$$

Table 2. *Observed structure amplitudes* (× 10) *and calculated structure factors*

Columns are headed by h, k and contain l, $\lvert F_o\rvert$ and F_c.

h	k	l	$\lvert F_o\rvert$	F_c
0	-10	2	142	-133
0	-8	1	406	-389
		2	60	53
0	-6	1	125	119
		2	62	57
		3	48	38
0	-4	1	131	-121
		2	89	86
		3	77	-75
0	-2	2	94	-98
0	2	1	165	156
		3	88	-92
0	4	0	53	-65
		1	253	269
		2	177	-169
		3	19	-21
0	6	0	195	-160
		1	258	-265
		2	84	-83
		3	134	141
0	8	0	178	-146
		1	259	-274
		2	138	113
		3	149	141
0	10	0	34	-30
		1	38	26
		2	124	123
		3	47	48
0	12	0	85	-80
		1	163	165
		2	54	43
		3	40	34
0	14	0	32	34
0	16	0	26	23
		1	27	-23
		2	29	31
0	20	0	23	-20
1	-11	2	127	-118
1	-10	1	181	183
		2	57	61
1	-9	2	30	-24
		3	45	46
1	-8	1	151	136
		2	28	22
		3	28	29
1	-7	1	170	-165
		2	26	17
		3	37	-31
1	-6	1	101	94
		2	30	28
		3	36	31
1	-5	1	15	-13
		2	110	95
		3	21	11
1	-4	1	162	-150
		2	88	-75
		3	18	-16
1	-3	1	752	648
		2	127	121
		3	65	68
1	-2	1	527	-472
		2	52	-45
		3	19	25
1	-1	1	267	-237
1	1	1	438	-408
		2	102	106
1	2	1	318	-261
		2	105	-100
		3	37	39
1	3	1	159	157
		2	69	-59
		3	55	-55
1	4	0	325	-347
		1	66	-61
		2	189	176
		3	48	-49
1	5	0	199	-179
		1	200	198
		2	182	-165
		3	26	30
1	6	0	104	-92
		1	157	153
		2	134	-130
		3	54	52
1	7	0	373	385
		1	52	-49
		2	154	130
		3	39	-42
1	8	0	165	-147
		1	246	-225
		2	143	142
		3	92	83
1	9	0	31	22
		1	82	84
		2	113	100
		3	90	82
1	10	0	167	-163
		1	112	109
		2	51	55
		3	153	157
1	11	0	87	-80
		1	55	55
		2	74	77
		3	29	-27
1	12	0	67	65
		1	28	-22
		2	165	173
1	13	1	141	125
		2	35	-30
		3	37	25
1	14	0	85	-85
		1	126	117
		2	52	-38
1	15	0	142	153
		2	49	37
		3	7	-5
1	16	0	26	6
		1	67	-55
		2	28	-15
1	18	0	35	-34
		1	22	-10
1	19	0	19	-11
2	-11	2	43	40
2	-10	1	184	182
		2	58	27
		3	18	-14
2	-9	1	37	38
		2	93	-92
		3	45	44
2	-8	1	21	12
		3	42	37
2	-7	1	20	20
		2	155	138
		3	39	31
2	-6	1	127	-112
		2	254	231
		3	50	44
2	-5	1	21	21
		2	60	-55
		3	119	118
2	-4	1	184	173
		2	313	-295
		3	13	-20
2	-3	1	135	113
		2	628	581
2	-2	1	810	-838
		2	309	-310
		3	29	-35
2	-1	1	1045	1193
		2	41	35
		3	59	70
2	0	1	347	-298
		2	165	168
		3	63	65
2	1	1	212	-211
		2	225	223
		3	114	130
2	2	1	28	24
		2	162	145
		3	53	51
2	3	0	153	-137
		1	101	95
		2	94	85
		3	34	-34
2	4	0	412	-399
		1	299	317
		2	110	104
		3	94	100
2	5	0	123	-116
		1	148	-148
		2	83	-84
2	6	0	209	206
		1	268	283
		2	117	107
		3	14	1
2	7	0	165	-175
		2	113	-102
		3	66	-64
2	8	0	323	297
		1	112	-117
		2	47	-44
		3	167	-156
2	9	0	132	129
		1	68	-88
		2	114	-106
		3	18	13
2	10	0	35	-34
		2	82	-89
		3	55	-46
2	11	1	104	-94
		2	44	40
2	12	1	63	57
2	13	0	126	-112
		3	22	-17
2	14	0	27	27
2	15	1	28	18
2	16	1	27	-26
2	17	0	44	43
		1	33	-30
		2	24	-14
2	18	0	22	-13
2	19	0	37	-34
		1	27	-18
3	-13	2	66	69
3	-12	1	64	-53
		2	54	-46
3	-11	1	77	77
		2	65	72
		3	47	57
3	-10	2	51	41
		3	18	-10
3	-9	1	37	25
3	-8	1	160	141
		2	151	-126
		3	27	-81
3	-7	1	104	-103
		2	28	30
		3	111	-115
3	-6	1	207	-192
		2	46	44
		3	131	125
3	-5	1	121	114
		2	295	-272
		3	155	-147
3	-4	1	144	125
		2	415	371
		3	106	105
3	-3	1	125	-114
		2	162	-168
		3	75	72
3	-2	1	120	114
		2	74	-79
3	-1	1	122	-115
		2	101	101
		3	97	-95
3	0	1	31	22
		2	73	-64
		3	20	-10
3	1	0	70	-72
		1	208	-210
		2	47	-44
		3	18	-15
3	2	0	374	395
		1	54	59
		2	81	75
3	3	0	29	-17
		1	230	235
		2	53	45
		3	133	130
3	4	0	98	94
		1	80	73
		2	110	110
		3	52	-39
3	5	0	46	36
		3	80	-76
3	6	0	124	-124
		1	247	242
		2	42	34
		3	52	53
3	7	1	37	-28
		2	61	-65
		3	69	64
3	8	0	76	71
		1	39	35
3	9	0	95	92
		1	161	-151
		2	51	41
		3	103	-94
3	10	0	52	51
		1	188	-187
		2	131	-132
		3	63	-57
3	11	0	238	-208
		1	27	-36
		2	227	-234
		3	49	49
3	12	0	145	-138
		1	123	-124
3	13	1	217	-231
		3	26	-25
3	14	0	66	-52
		1	61	55
3	15	0	141	-152
		1	28	-22
3	19	1	30	29
4	-14	2	52	52
4	-13	1	71	73
		2	62	61
4	-12	1	149	145
4	-11	1	42	37
		2	56	70
		3	27	23
4	-10	1	69	-77
		3	28	29
4	-9	1	88	81
		2	32	20
4	-8	1	43	41
		2	85	74
		3	59	-47
4	-7	1	53	59
		2	101	-94
		3	59	50
4	-6	1	141	-117
		2	81	-72
		3	51	-50
4	-5	1	439	-417
		2	101	-83
		3	227	-222
4	-4	1	18	21
		2	58	-55
		3	225	214
4	-3	1	37	45
		2	438	-387
		3	59	-55
4	-2	1	214	-209
		2	182	161
		3	35	32
4	-1	1	124	-132
		2	71	67
		3	36	34
4	0	0	272	-251
		1	67	68
		2	59	51
		3	31	-25
4	1	0	238	-251
		1	38	-33
		2	105	-95
		3	32	-25
4	2	0	72	73
		1	215	-216
		2	163	-172
		3	65	-73
4	3	0	195	197
		1	342	-360
		2	37	40
		3	113	114
4	4	0	460	-450
		1	131	-122
		2	222	-225
		3	26	-29
4	5	0	222	-212
		1	84	92
		2	94	87
4	6	0	69	60
		1	181	-182
		2	44	-35
		3	74	-69
4	7	0	52	-50
		1	85	84
		2	84	-83
4	8	0	144	-132
		1	82	-90
		3	18	-13
4	9	0	152	138
		2	157	135
		3	30	27
4	10	0	54	56
		1	107	110
4	11	0	39	-44
		1	101	101
		2	65	75
		3	17	13
4	12	0	64	69
		1	44	-45
		2	54	-63
4	13	0	41	-39
		3	21	14
4	14	0	66	-72
		1	60	-45
		2	50	33
4	15	0	26	-24
4	19	0	17	22
5	-14	1	154	-147
		2	60	-92
5	-13	2	92	-92
5	-12	1	170	-150
5	-11	1	43	-32
		2	63	-63
		3	27	23
5	-10	1	47	47
		2	53	-53
5	-9	1	111	-107
		3	38	-30
5	-8	1	15	-13
5	-7	1	117	-75
		2	48	39
		3	68	72
5	-6	1	157	136
		2	117	106
		3	146	-140
5	-5	1	32	25
		2	35	30
		3	112	115
5	-4	1	75	63
		2	41	-31
		3	17	-15
5	-3	1	72	-75
		3	39	36
5	-2	1	52	-44
		2	149	-150
		3	16	14
5	-1	1	43	-41
		2	67	65
		3	16	14
5	0	1	135	-128
		2	25	-27
5	1	0	38	-42
		1	65	-54
		2	69	91
		3	86	-74
5	2	0	276	247
		1	45	45
		2	114	108
		3	56	-41
5	3	0	145	-139
		1	82	95
		2	125	-128
		3	29	27
5	4	0	166	-152
		1	86	-72
		2	132	-129
		3	42	37
5	5	0	94	-94
		1	239	-212
		2	29	-25
		3	69	60
5	6	0	183	-190
		1	22	-13
		2	58	68
		3	40	-41
5	7	0	160	-179
		1	65	-69
		2	78	79
		3	100	-94
5	8	2	91	-89
5	9	1	61	55
		2	94	-91
5	10	0	32	37
		1	111	-103
		2	95	82
		3	65	-75
5	11	0	26	20
		2	54	56
		3	24	20
5	12	0	150	145
		1	211	214
		2	95	-101
		3	27	-23
5	13	0	45	50
		1	126	127
		3	22	-18
5	14	0	161	155
		1	59	-57
		3	19	-19
5	15	0	92	88
5	16	0	35	-35
		1	25	20
5	18	0	19	17
		1	16	-24
5	19	0	23	21
6	-12	1	66	-72
		2	54	-52
		3	15	18
6	-11	1	66	-75
		2	69	-78
		3	36	34
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		3	105	-103
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		3	85	67
6	5	0	103	102
		1	175	162
		2	154	-150
		3	47	45
6	6	0	48	-46
		1	24	-25
		2	85	52
		3	26	27
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		1	107	-105
		2	42	47
6	8	0	23	-19
		1	26	24
6	9	0	106	102
		1	159	163
		2	66	66
		3	66	-68
6	10	0	122	-110
		1	126	-125
		3	63	54
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		2	62	-54
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6	16	0	23	17
6	17	0	21	-18
		1	46	-36
6	19	0	12	-12
7	-14	1	42	37
		2	53	58
7	-12	1	71	70
		3	46	-42
7	-11	1	67	70
		3	16	-15
7	-10	1	44	48
		2	66	-60
		3	17	6
7	-9	1	61	67
		2	44	57
		3	31	37
7	-8	1	134	-116
		2	53	-51
		3	26	-22
7	-7	1	115	98
		2	82	68
		3	18	-15
7	-6	1	65	-84
		3	24	-29
7	-5	2	50	30
		3	28	19
7	-4	3	68	69
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		2	55	-51
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		3	63	-43
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		3	93	-81
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		1	94	-84
		2	31	27
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7	4	1	97	90
		2	112	-105
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		1	42	-40
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		3	46	-34
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		1	44	54
		2	63	62
		3	24	23
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		1	109	100
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		1	63	57
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7	16	0	26	37
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		2	54	-57
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		2	46	50
		3	31	-29
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		3	11	-12
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		1	42	35
		2	44	-33
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		1	26	-25
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		2	11	14
14	-7	1	49	-46
		2	35	-39
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		2	30	42
14	-4	1	42	-42
		2	25	-32
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		2	25	21
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		2	22	-24
14	8	1	19	-5
14	11	0	13	-10
15	-5	1	36	-40
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15	-3	2	34	41
15	-2	1	22	15
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		2	19	21
15	0	2	19	14
15	1	1	34	31
		2	19	17
15	2	0	27	-27
		2	28	-29
15	3	0	29	34
		2	26	29
15	4	0	20	18
		1	35	5
15	5	0	19	21
		1	33	33
15	6	1	57	32
15	7	0	17	19
15	8	0	15	18
16	-2	1	16	-14
16	-1	1	16	-15
16	0	1	16	17
16	1	1	16	12
15	5	0	22	-21

indicate that only the sign obtained for 710 ($E=1{\cdot}34$) was wrong. Table 3 defines the thermal motion ellipsoids and Fig. 1 shows bonded distances and inter-bond angles.

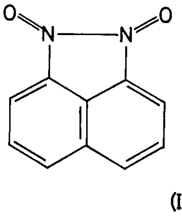
Table 3. 1,8-*Dinitrosonaphthalene*

Vibrational ellipsoids; u^2 is along the principal axes of the atomic ellipsoids of vibration and direction cosines are with respect to the crystallographic axes

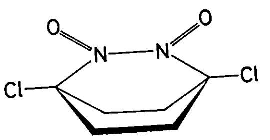
	u^2 (Å)^2	Direction cosines *a*	 *b*	 *c*
C(1)	0·223	0·616	−0·785	−0·032
	0·215	0·393	0·382	−0·854
	0·184	−0·683	−0·488	−0·520
C(2)	0·245	0·520	−0·288	−0·790
	0·232	−0·287	0·827	−0·521
	0·209	0·804	0·483	0·324
C(3)	0·261	0·811	0·060	−0·584
	0·234	−0·546	0·432	−0·737
	0·205	0·209	0·900	0·341
C(4)	0·263	0·732	0·606	−0·339
	0·221	0·632	−0·437	0·660
	0·191	−0·252	0·665	0·671
C(5)	0·285	0·046	0·961	0·227
	0·271	−0·050	−0·271	0·973
	0·211	0·998	−0·058	0·039
C(6)	0·295	0·319	−0·810	−0·454
	0·234	−0·080	0·494	−0·888
	0·221	0·945	0·315	0·078
C(7)	0·274	0·632	−0·503	−0·565
	0·226	0·570	0·817	−0·126
	0·214	−0·526	0·280	−0·815
C(8)	0·226	0·892	0·234	−0·397
	0·210	−0·095	0·934	0·301
	0·186	−0·442	0·271	−0·867
C(9)	0·219	0·645	0·664	−0·409
	0·209	−0·566	0·747	0·313
	0·175	−0·514	0·011	−0·857
C(10)	0·249	0·439	0·898	−0·036
	0·213	0·829	−0·402	−0·370
	0·185	0·347	−0·176	0·928
N(1)	0·252	0·474	0·872	−0·161
	0·239	0·263	−0·011	0·964
	0·197	0·840	−0·489	−0·212
N(2)	0·256	0·521	0·493	−0·719
	0·223	0·813	−0·036	0·583
	0·195	0·262	0·869	0·378
O(1)	0·312	0·450	0·290	0·830
	0·287	−0·116	−0·919	0·420
	0·188	0·885	−0·267	−0·367
O(2)	0·329	0·616	0·568	−0·573
	0·279	0·740	−0·179	0·656
	0·193	−0·270	0·804	0·491

Results and discussion

The crystal is built up of isolated monomeric molecules of the form (I). An internal 'nitroso dimer' of this type has been proposed for 2,2′-dinitrosodiphenyl and a similar system has been found for 1,4-dichloro-1,4-dinitrosocyclohexane (II) (Hodgkin, 1969).



(I)



(II)

In order to take up the 'nitroso-dimer' form the 1,8-dinitrosonaphthalene molecule is considerably distorted. In Fig. 1 the maximum e.s.d. found in bond length for bonds not involving hydrogen atoms is 0·008 Å and for similar interbond angles 0·4°. Differences in bond length of 0·025 Å and in bond angle of 1·2° must be regarded as significant. To the first approximation the molecule is planar with the molecular plane roughly parallel to the (001) crystal plane (Fig. 2); the equation to the least-squares best molecular plane is $2{\cdot}513x/a - 3{\cdot}291y/b + 1{\cdot}926z/c = 1$. The molecule has a mirror plane at right angles to the molecular plane and passing through C(9) and C(10). Any observable deviations from mirror symmetry are well within experimental error. Whilst deviations from the molecular plane are small they are between 2·5 and 15 times larger than the e.s.d.'s of the *z* parameters of the atoms in question. Moreover, if it is accepted that the deviations from the molecular plane are significant then the mirror plane perpendicular to it through C(9) and C(10) is still preserved. The naphthalene molecule has a third mirror perpendicular to both the molecular plane and the C(9)–C(10) vector. This plane is lost in the naphthalene nucleus of 1,8-dinitrosonaphthalene. The angular distortions are well outside experimental error and lengths of the bonds C(1)–C(9) and C(9)–C(8) are significantly different from those of C(4)–C(10) and C(10)–C(5), and from the observed lengths of the corresponding bonds in naphthalene (Cruickshank, 1957). The other carbon–carbon bond lengths in 1,8-dinitrosonaphthalene are in good agreement with those in naphthalene. The distortions in bond angle in the naphthalene nucleus of 1,8-dinitrosonaphthalene are exactly those that are to be expected if the molecule is imagined as a semi-rigid frame with 120° angles which is distorted by pulling together N(1) and N(2). Remarkably similar distortions are found in acenaphthenequinone(III) (Mak & Trot-

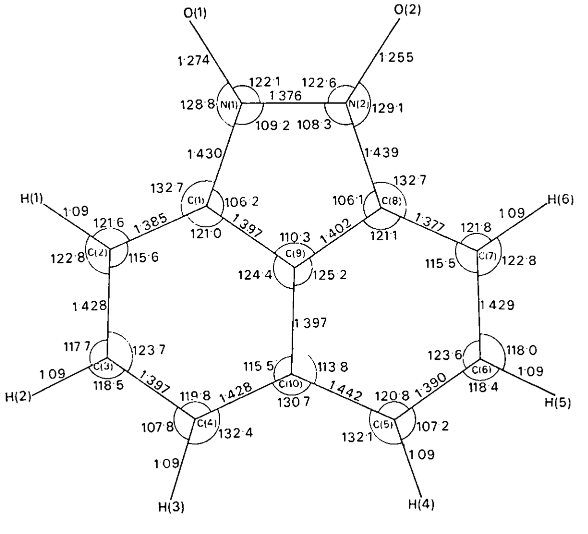
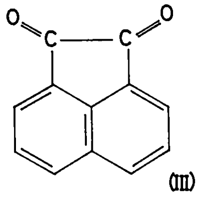


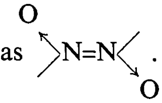
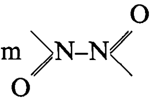
Fig. 1. Molecular bonds and angles.

ter, 1963) and *cis*-1,2-acenaphthenediol (Trotter & Mak 1963) but in neither structure were the differences between the C(1)–C(9), C(9)–C(8) and C(4)–C(10), C(10)–C(5) bond lengths observed, perhaps due to the higher experimental error of these determinations.



(III)

The nitrogen–carbon bonds (1·435 Å av.) are shorter than the aromatic C–NO$_2$ bonds (1·47 Å) the same length as the C–N bond in PhN=COEt.

The best published determination of a nitroso dimer is that of the aliphatic 2-nitronitrosoethane (Boer & Turley, 1967). In 1,8-dinitrosonaphthalene the C–N bonds C(1)–N(1) and C(8)–N(2) are shorter (1·437 Å av.) than those in 2-nitronitrosoethane (1·462 Å). They are shorter than the C–NO$_2$ bond (1·47 Å) (Allison, Bailey & Prout, 1970) in aromatic nitro compounds, the same length as the C–N bond in PhNHCOMe (1·435 Å) (Brown & Corbridge, 1954) and much longer than the C=N in furoxans (Cady, Larson & Cromer, 1966). The bond length suggests they are essentially σ bonds and the difference between these and the C–N bond of 2-nitronitrosoethane is no greater than might be expected from the proposed difference in covalent radii for carbon atoms using sp^3 and sp^2 hybridization. The bond N(1)–N(2) (1·367 Å) is little shorter than the N–N single bond in diacetylhydrazine (1·396 Å) (Shintani, 1960) and is very significantly longer than the equivalent bond in 2-nitronitrosoethane (1·315 Å). The N–O bonds N(1)–O(1) and N(2)–O(2) (1·264 Å av.) are not significantly longer than those in 2-nitronitroso-ethane. Boer & Turley (1967) concluded that in 2-nitronitrosoethane the most important resonance form was  $\overset{O}{\rangle N}{=}\underset{O}{N\langle}$. The N(1)–N(2) bond length suggests that in 1,8-dinitrosonaphthalene the contribution from this form is greatly reduced and either the resonance form  $\underset{O}{\rangle N}{-}\overset{O}{N\langle}$ is of increased importance or the nitrogen atoms are involved in a π bonding interaction with the naphthalene nucleus. The latter is unlikely because of the N–C bond length and the impossibility of writing plausible resonance forms. If a π bonding interaction did occur it would most probably involve the naphthalene π^* orbitals and would tend to stabilize an unstrained furoxan-like form. The former alternative requires the N–O bonds in 1,8-dinitrosonaphthalene to be shorter than those in 2-nitronitrosoethane, when in fact they are longer. The short N–O bonds are a major problem in Boer & Turley's model for 2-nitronitrosoethane. Detailed interpretation of the electronic structure of 1,8-dinitrosonaphthalene must await a rigorous treatment.

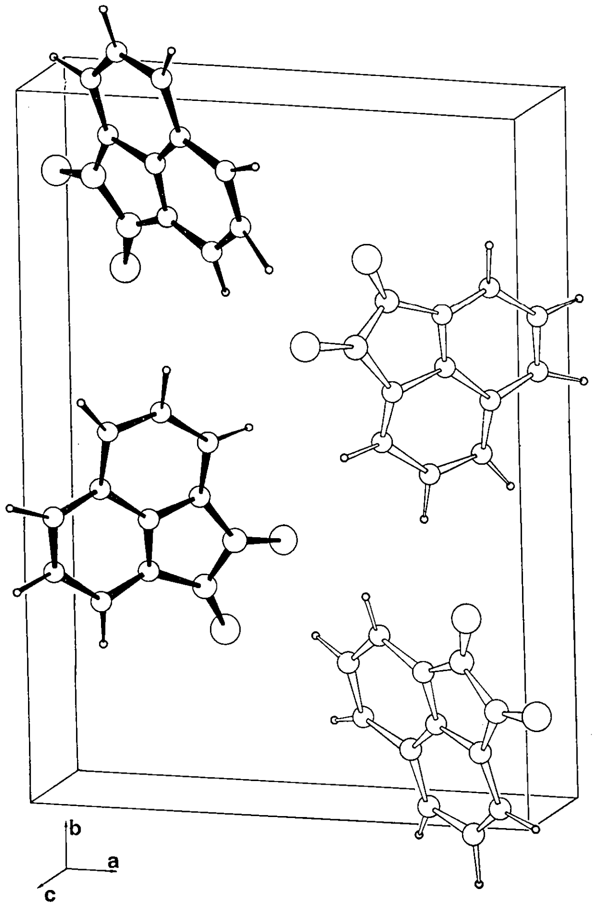


Fig. 2. Clinographic projection, along the *z* axis, of the content of one unit cell.

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